

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Analytical method of ^{14}C in liquid effluent of nuclear power
plant -- Wet oxidation**

核动力厂液态流出物中 ^{14}C 分析方法—湿法氧化法

(English Translation)

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1 National Environmental Protection Standard of the People's Republic of China

Analytical method of ^{14}C in liquid effluent from nuclear power plant-Wet oxidation Analytical method of ^{14}C in liquid effluent of nuclear power plant

--Wet oxidation

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foreword in order to implement the "Environmental Protection Law of the People's Republic of China" Republic Nuclear Safety Law, which regulates the analysis method of ^{14}C in liquid effluent from nuclear power plants, and formulated this standard.

This standard specifies the ^{14}C analysis method-wet oxidation method for liquid effluent from nuclear power plants.

This standard is issued for the first time.

This standard is formulated by the Department of Nuclear Environment Safety Supervision, Regulations and Standards of the Ministry of Ecology and Environment.

2 Normative references

The content of this standard refers to the following documents or clauses therein. For undated references, the valid version is Used in this standard.

GB/T 6682 Analytical laboratory water specifications and test methods GB 11217 General requirements for monitoring effluent from nuclear facilities HJ 493 Technical regulations for the storage and management of water samples

3.1 Methodology

Treatment of water samples by acid hydrolysis scrubbing, adding persulfate oxidant (catalyst may be added as needed) Adding phosphoric acid and sodium persulfate to the water sample and heating to convert the inorganic and organic carbon contained in the water sample into carbon dioxide.

The generated carbon dioxide is purged with a carrier gas (nitrogen) and then absorbed with an inorganic lye or an organic lye 1. CO₂ generated After collecting the liquid and adding scintillation liquid to make a liquid scintillation test sample, a liquid scintillation counter was used to measure the activity of ¹⁴C.

3.2 Conversion of carbon dioxide gas

Conversion of inorganic carbon to carbon dioxide.

CO₃²⁻ + 2H⁺ → CO₂ + H₂O

HCO₃⁻ + H⁺ → CO₂ + H₂O

H₂CO₃ → CO₂ + H₂O

Conversion of organic carbon to carbon dioxide.

Organic carbon S₂O₈²⁻ + H₂O heating % 00% 00% 00 2H₂SO₄ - CO₂

3.3 Absorption of carbon dioxide gas

Inorganic base. CO₂ + 2OH⁻ → CO₃²⁻ + H₂O Organic base (take HO(CH₂)₂NH₂ as an example). HO(CH₂)₂NH₂ + CO₂ → HO(CH₂)₂NH₂ · CO₂ when selecting organic lye, consideration should be given to the compatibility with the scintillator.

24 Reagent

Unless otherwise specified, the reagents used in this standard use analytical and purification reagents that meet national standards.

The water is not lower than GB/T 6682 secondary water or water of equivalent purity.

4.1 Sodium persulfate solution. 100g/L.

4.2 Phosphoric acid solution. The volume fraction is 5%.

4.3 Alkali absorption solution. 1mol/L sodium hydroxide solution, or organic lye.

4.4 ¹⁴C reference materials. ¹⁴C labeled n-hexadecane and Na²¹⁴CO₃.

4.5 Toluene (C₆H₅CH₃).

4.6 Triton X-100 [C₈H₁₇ (C₆H₄) (OCH₂CH₂)₁₀OH].

4.7 2,5-Diphenyloxazole [OC (C₆H₅) = NCH = CC₆H₅]. PPO for short, pure flicker.

4.8 1,4- [Bis- (5-phenyloxazole-2)] benzene, ([OC (C₆H₅) = CHN = C] 2C₆H₄). referred to as POPOP, flickering pure.

4.9 scintillation fluid.

-Use toluene (4.5) and Triton X-100 (4.6) to mix at a ratio of 2 1. Weigh out 4.00g of PPO (4.7) and 0.30g POPOP (4.8) were dissolved in 1000 ml toluene-triton X-100 mixed solution and transferred to brown reagent Store the bottles juxtaposed in a dark place. The scintillation fluid should be used immediately.

-You can also use the commercial scintillator directly.

5 Equipment and materials

5.1 For an example of a sample preparation device, see Appendix A. The total organic carbon analyzer 2 can also be used as a sample preparation device.

5.2 Analytical balance. The accuracy is 0.1mg.

5.3 Brown glass bottle. 1L, used to store water samples, need to be dried before use.

5.4 Pipette. 1ml, 5ml graduated pipette.

5.5 High-purity nitrogen. purity ≥99.99%.

5.6 Liquid flash counting bottle. 20ml.

5.7 Low background liquid scintillation counter. background ≤ 2cpm.

5.8 Glass absorption bottle. 50ml or 100ml.

6 Sample collection and storage

6.1 The collection and storage of samples shall be performed in accordance with the relevant regulations of HJ 493 and GB 11217.

6.2 When collecting samples, the samples should be filled in brown glass bottles and filled, leaving no headspace, and protected from light.

6.3 The collected water samples shall be determined within 24 hours.

7 Analysis steps

7.1 Clean the sample preparation device with pure water for later use.

7.2 Check the air tightness of the sample preparation device (5.1) by introducing nitrogen.

7.3 Continue to introduce nitrogen (5.5) at a flow rate of 30 ml/min to 50 ml/min until the preparation of the carbon dioxide absorption solution is completed.

Optional commercial TOC analyzer.

37.4 Add 20 ml of alkaline absorption solution (4.3) to a glass absorption bottle (5.8).

7.5 Add 60.0ml (V1) water sample to the reactor. If suspended matter exists in the water sample to be tested, first use a 0.45μm filter membrane Filter.

7.6 Add 20 ml of phosphoric acid solution (4.2) and 20 ml of sodium persulfate solution (4.1) to the reactor.

7.7 Heat the liquid in the reactor and control the temperature at 95 ± 3 ° C to maintain a sufficient reaction time³.

7.8 Purge the carbon dioxide gas generated during the reaction with nitrogen, and dry the alkali absorption into the glass absorption bottle.

The liquid was collected to produce a carbon dioxide absorbing solution (V2).

7.9 Transfer 8.0ml (V3) carbon dioxide absorption liquid to the liquid scintillation counting bottle (5.6), add 12.0ml scintillation liquid (4.9), Tighten the bottle cap and shake well to make the sample 4 to be tested. The samples to be tested should be kept away from light.

7.10 Replace the test water with a calibration water sample prepared with a ¹⁴C reference material and having an activity concentration close to the ¹⁴C activity concentration in the effluent.

In accordance with the methods and steps of 7.1 to 7.9 above, prepare a calibration sample.

7.11 Use deionized water as a blank water sample instead of the water sample to be tested, and prepare blank samples according to the methods and procedures of 7.1 to 7.9 above.

7.12 Use a low background liquid scintillation counter to measure the test sample (7.9), the